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The composite sample at creameries. Size of samples; Chromate preservatives.

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THE COMPOSITE SAMPLE AT CREAMERIES.

SIZE OF SAMPLES; CHROMATE PRESERVATIVES.

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I. Size of Daily Samples.

When, early in 1890, the writer first proposed the composite sample—or as it is now generally called, improperly, the "composite test"—as a means of rendering practicable the "value plan" at creameries, he was particular to emphasize, by repetition, the statement that to insure accuracy "the daily samples must be, for each patron, proportionate in amount to his daily deliveries" (Bulletin No. 9). Without this precantion it is evident that the composite may fail of being what it is intended, namely, a miniature of the entire delivery of the patron for the period.

In the directions for carrying out the plan (same Bulletin) it was suggested that a convenient size for daily samples would be, in most cases, one-tenth as many cubic centimeters as the number of pounds in the delivery; and it was proposed to measure the samples in a graduated cylinder. This, the reader must bear in mind, was in the very infancy of the value system; we have now a much easier way of taking proportionate samples, which will be mentioned further on.

Some months after the publication of Bulletin No. 9, Prof. W. W. Cooke, in the Vermont Station annual report for 1890, after referring to the great stress that had been laid "by some people" on the taking of samples proportionate to the deliveries and the fact that this was being practiced in Iowa, attempted to prove that in practice this precaution is entirely unnecessary, while of course freely admitting its correctness in theory. He based his conclusions upon an experiment. For four consecutive days he analyzed the daily deliveries of eighteen patrons of a certain creamery,

and calculated the butter-fat actually delivered by each, in comparison with what a composite, made up of daily samples all of the same size, would have shown. The greatest difference found was .67 of a pound of fat, the least .01 of a pound—the former on a total delivery of 111 pounds of butter-fat. Neither the weights of the daily deliveries nor the fat percentages are given, but both were said to be quite variable. From these results Prof. Cooke concludes that "the error introduced into the work by taking all the samples of the same size is much too small to be of any account," and that "the taking of samples varying in size with the quantity of milk, is a needless precaution." These are certainly sweeping conclusions to draw from a single experiment; it would have been more logical, it seems to the present writer, to limit them to that particular experiment or to cases where only natural variations in the quality of the milk occur. Where only such variations occur Prof. Cooke's conclusions are entirely valid, and have been so recognized in practice. Nearly all creameries that pay by the test, acting on the assumption that most men are honest, have adopted his plan and take the samples all of like size; and the present writer has frequently said of this plan that it is "usually, nearly enough correct." But usually is not always; and it is possible for cases to occur in which the error will be considerable. If a patron brings a number of small deliveries of very rich milk and a number of large deliveries of comparatively poor milk, the composite sample composed of like sized daily samples will give him credit for more fat than he has delivered. Thus, suppose on six days he brings 200 pounds per day of three per cent milk, and on six days 100 pounds per day of milk enriched to seven per cent fat,—the composite being tested at the end of the fortnight.

1200 pounds × .03 equals 36 pounds fat.
600 pounds × .07 equals 42 pounds fat.

78 pounds of fat actually delivered.

The composite sample, obtained as above described, will test five per cent (the mean of three and seven). Therefore, 1,800 pounds × .05 = 90 pounds of fat, credited to the patron.

Here are twelve pounds of fat paid for that were never delivered; the patron has furnished only lawful milk—assuming

it to be in Iowa, where the standard is three per cent; the creamery has simply, by its own method, wronged itself. At least that is what the patron would say; and, in truth, when a creamery adopts the value plan it does in substance say to its patrons something like this: "Bring as rich or as poor milk as you please, above the legal standard, in any amounts you please; we will value it and pay you accordingly."

It will be understood, of course, that no such error as the above can occur when the daily samples are taken as originally directed by the writer, *i. e.* for each patron, proportional in amount to his daily deliveries.

The practical question puts itself: To avoid this danger, must then every daily sample taken at the creamery be measured? The writer offers reply as follows: Where a patron's deliveries run fairly uniform in amount, from beginning to end of composite period, the usual method now in vogue is nearly enough correct; but where a patron's deliveries show wide variation in amount, within the time of a composite period, the daily samples should unquestionably be taken proportionate in amount to the deliveries; then, however great the variation in richness, the composite will tell only the truth.

The original mode of taking proportionate samples—measuring in a graduated cylinder—is too slow. There is now a better way, namely, by means of a sampling tube. This is plunged (not too rapidly) to the bottom of the weigh-can, when by a simple device its lower end is closed, and it is then withdrawn, holding a vertical section of the milk in the weigh-can; which vertical section will of necessity be proportional in amount to the different deliveries. Several forms of sampling tube have been described, but the best one known to the writer is that recently devised by Prof. Scovell, of the Kentucky Station, and used in the dairy work at the World's Fair.

Should this or any other device for taking proportionate samples be found as convenient, or nearly as convenient, as the dipper method of taking uniform samples, it is to be hoped that the former will entirely replace the latter, and thus obviate an ever threatening source of error in the present mode of carrying out the value plan. Should this sug-

gestion be adopted, quart jars for composites will be found preferable to pints, at least for patrons making large deliveries. In the writer's experience the Lightning jar has been found more convenient than the Mason.

II. Chromate Preservatives.

Toward the end of the summer of 1892, the writer, having been long searching for a substitute for mercuric chloride (corrosive sublimate), observed the interesting fact that potassium bi-chromate possesses in a high degree the power to keep milk from curdling, and, as time allowed, he made experiments to see if the Babcock test would prove the preservation to be complete, and whether the chromate would interfere with the working of the test. The first experiments were upon two week composites, and the results were quite satisfactory; so much so that at the meeting of the State Dairy Association, held at Ames, November, 1892, he mentioned the discovery as one promising much value, but expressly refrained from recommending it for use in creameries until he should have time for further trials of it. At this time the writer supposed he was the first to observe this remarkable property of the bi-chromates, but later his attention was called to an article in Biedermann's *Central-Blatt f. Agrikultur-chemie*, August, 1892, giving an account of the very same discovery by a Swedish chemist, J. A. Alèn. Alèn, therefore, has priority in the discovery. The Swedish chemist in his experiments used daily milk samples of only 10 c. c.; and, *holding the composites at a temperature not higher than 59 deg. F.*, found .5 gramme (7½ grains) of the bi-chromate sufficient to keep in good condition composites of 30 and even 60 day periods, having a total volume of 250 to 500 c. c. (8½ to 17 fluid ozs). But, holding the composites at higher temperatures, 68 deg. F. or above, his results were not satisfactory, the tests coming too low—partly, he explains, because of actual destruction of fat, with liberation of fat acids, but chiefly because of the formation of cream-lumps which refuse to be re-emulsified by rotation of the jars. His tests were made with the Swedish milk-test, the Lactocrite.

In the writer's earliest experiments he was misled into the belief, as stated before the Dairy Association, that the use of bi-chromate would necessitate an increased charge of acid in operating the Babcock test to yield correct results; but this was soon found to be a mistake, occasioned by the faulty working of a loose-belted Babcock machine.

The writer's experiments with chromate preservatives have now (November, 1893) been carried on with only slight intermissions for over a year; the trials of different mixtures and amounts of preservatives are numbered by scores and the separate "tests" by hundreds; the experimental record would cover many pages of this Bulletin and might only confuse the reader; therefore it is thought best to omit this record almost entirely, and report only the general results and conclusions, with a few numerical data for illustration.

The earlier systematic experiments (made during the winter of 1892) disclosed the fact that while potassium bi-chromate, in doses of ten to twenty grains—to 250 or 300 c. c. of milk—will preserve the latter perfectly for two weeks, if the time is prolonged there frequently sets in, usually during the third or fourth week, a peculiar fermentation giving rise to a distinct fruity odor, doubtless from the formation of butyric ether or ethers, accompanied by a destruction of butter-fat that often reduces the test .3 or .4 per cent in a few days. At the same time, or a few days later, portions of the cream gather into compact masses, as mentioned by Alèn, that refuse to be broken up by rotation. These masses, reminding one of islands, are probably "colonies" of the organisms that destroy the fat and produce the fruity aroma.

The bi-chromate, then, under certain conditions, actually promotes the destruction of milk-fat after a certain length of time. This is a serious fault in a substance to be used for preserving milk samples. It is true that with ordinary amounts of the preservative (15 to 25 grains in a jar) this disastrous effect does not usually appear during the first fortnight, and therefore it may be safely used for two week composites; but it seems desirable that a preservative shall be efficient for a full month; unless it is, it does not effect the greatest possible saving in labor. The old preservative, cor-

rosive sublimate, in doses of 15 to 25 grains per jar, comes up to this requirement; but, beside being very poisonous, requiring therefore the admixture of some color as a danger signal, it is rather expensive, and therefore for three years it has been the aim of the writer to find something less objectionable that would be equally or nearly as efficient. To this end he instituted experiments aiming to overcome, by suitable additions, the peculiar weakness or failing of the bi-chromate.

First, on the supposition that the acidity of the bi-chromate might be the cause of its bad behaviour, addition of alkalies was tried—sodium carbonate, caustic soda, and slaked lime. It was soon found, however, that these aggravated the difficulty, especially the caustic soda and lime, the latter in a marked degree; they hastened the development of the fruity odor and the destruction of butter-fat. Abandoning these, small additions of the "old stand-by," corrosive sublimate, were next tried and with the most favorable results. It was found that an addition to the bi-chromate of one-eighth its weight of corrosive sublimate fully corrects the fault, entirely preventing the peculiar fat-destroying fermentation that is induced by the chromate when used alone; this being true for monthly periods and longer,—daily samples being 15 c. c., total volume of composite being 300 to 400 c. c., and the amount of preservative mixture 15 to 25 grains.

If the proportion of corrosive sublimate in the mixture be increased to one-fourth the weight of the bi-chromate, a somewhat smaller dose of the powder suffices, viz.: 10 to 15 grains; but the sublimate should not be increased above that proportion, for it will then produce a "flecky" condition in the composite, this being the peculiar fault of corrosive sublimate. As small a proportion as one to sixteen (of sublimate to bi-chromate) has given good results, in the cooler months, but I cannot recommend it since in a few cases there appeared signs that the fat-destroying action was setting in.

With daily samples of 15 c. c. (one-half a fluid ounce), I can recommend the following mixtures and amounts, for monthly composites:

Potassium bi-chromate, powdered, 16 ounces.

Mercuric chloride (corrosive sublimate), powdered, 2 ounces.

Well mixed; 15 to 25 grains in each jar.

Or—

Potassium bi-chromate, powdered, 16 ounces.

Mercuric chloride (corrosive sublimate), powdered, 4 ounces.

Well mixed; 10 to 15 grains in each jar.

As usual, at each addition of the daily samples the jar must be rotated (not shaken with a churning motion) until the cream is all washed from the sides of the jar and redistributed in the milk; the jar must therefore not be moved, unless to *thoroughly* rotate it, until the next daily sample is added. Either of the above described mixtures is a more efficient preservative than even pure corrosive sublimate, and vastly more so than the pure bi-chromate. But this is not quite the whole story. Every rose has its thorn, and these powders have their fault,—a fault belonging to the bi-chromate—namely, that they are quite liable to give (black) muddy tests (by the Babcock) if the temperature of the composites (and acid) at testing is much above 60 degrees Fahrenheit, or if the acid is too strong. But with the conditions right—acid 1.82 to 1.83 sp. gr. and temperature not too high—there is usually no difficulty in getting clear tests, correct to within $\frac{1}{10}$ per cent; this is true at least for all but the hottest part of the summer and will probably prove the same for that period. This liability to bring black, muddy tests does not pertain in any degree to corrosive sublimate when used alone, which is a large point in its favor.

It must further be said that with any preservative yet tried there is a tendency for the tests of monthly composites to come a *trifle* below the true mean of the daily samples, especially if the conditions of temperature and strength of acid are not exactly right; thus they frequently, but by no means always, come about $\frac{1}{10}$ per cent below the true mean. This the writer at present thinks is more liable to occur with the bi-chromate mixtures than with the pure corrosive sublimate, but he is not quite certain of it. Even if the deficit does sometimes occur, it is only a small error, scarcely more

than is liable to occur with fresh milk in ordinary routine testing and should not, in the writer's opinion, prevent the adoption of the monthly composite plan.

Finally, in justice to the pure bi-chromate preservative, the writer must add that while he has repeatedly found forty grains insufficient for monthly composites, he has in two trials (the only ones where such large amounts were used) had passable success with sixty and eighty grains respectively.

To illustrate the above remarks a few of the numerous experimental results obtained are inserted here:

Monthly composite—August 18th to Sept. 18th, 1893. Milk for eleven days fresh from college barn; for balance of month creamery milk, as delivered by patrons.

Daily samples, 15 c. c. each.

Tests of 27 daily samples—mean, 3.63 per cent fat.

Tests of composite samples, Sept. 18th to 20th.

		Condition of sample.	
Corrosive sublimate, (*pure*).	15 grains.	Good.	3.60 3.55
	25 grains.	Good.	3.60 3.60
	40 grains.	Good.	3.60 3.55
Potassium bi-chromate, (*pure*).	15 grains.	Bad. Lumpy.	3.15 3.15
	25 grains.	Lumpy.	? muddy. ? muddy.
	40 grains.	Lumpy.	2.8 muddy. 3. muddy.
Potassium bi-chromate and corrosive sublimate, 16:2.	10 grains.	Fair.	? muddy. 3.60 3 50
	15 grains.	Good.	3.65 3.60 ? muddy 3.50
	25 grains.	Good.	3.55 ? muddy. 3.60 3.50
Potassium bi-chromate and corrosive sublimate, 16:4.	10 grains.	Good.	3.50 3.60 3.55
	15 grains.	Good.	3 55 3.60 muddy. 3.50 3.60
	25 grains.	Some flecky	3.60 little mud. 3.45 3.50 3.60 little mud.

NOTE—*Acids of sp. gr.* 1.82 *and* 1.83 *were both tried. That of* 1.83, *in a warm room, often gave muddy tests; that of* 1.82, *in the cool part of the day, brought the lower results.*

Monthly composite—August 25th to September 25th. All creamery milk, as delivered by patrons.
Daily samples. 15 c. c. each.
Tests of 26 daily samples—mean, 3.93 per cent fat.
Tests of composite samples, October 25th to 27th.

		Condition of sample.	
Corrosive sublimate, (*plus aniline red to color*).	15 grains.	Good.	3.85 3.90 3.95 3.90
	25 grains.	Flecky.	3.85 3.75 4.00 3.80
	40 grains.	Flecky.	3.85 3.90 3.90 3.85 3.80
Potassium bi-chromate, (*pure*).	25 grains	Bad.	3.50
		Lumpy.	3.40
	40 grains.	Bad.	———
		Can not measure.	———
	60 grains.	Good.	3.80 3.75 3.70 3.70 3.80 ? muddy. ? muddy.
	80 grains.	Good.	3.80 3.75 3.80 ? muddy.
Potassium bi-chromate and corrosive sublimate, 16:2.	15 grains.	Good.	3.80 3.75 3.85 4.00 3.90 3.80
	25 grains.	Some flecky.	3.90 3.90 3.85 3.90 3.85 3.70
Potassium bi-chromate and corrosive sublimate, 16:4.	15 grains.	Good.	3.75 3.90 3.80 3.85 3.85
	25 grains.	Too flecky.	3.80 3.75 ? muddy.

NOTE—*Acids of 1.82 and 1.83 sp. gr. used. The former gave the lower results; the latter the higher, and in warm room the muddy tests.*

Monthly composite—September 19th to October 19th. **All creamery milk, as delivered by patrons.**

Daily samples 15 c. c. each.

Tests of twenty-nine daily samples—mean, 4.30 per cent fat.

Tests of composite samples, October 20th and 21st.

		Condition of sample.	Sp. gr. of Acid.
Corrosive sublimate, (*plus aniline red to color*).	10 grains.	Curdled.	.....
	15 grains.	Good.	4.20 / 4.10 — 1.82
			4.35 / 4.20 — 1.83
	25 grains.	Flecky.	4.30 / 4.30 — 1.82
			4.20 / 4.35 — 1.83
Potassium bi-chromate, (*pure*).	10 grains.	Curdled.	.....
	15 grains.	Very bad, cannot measure.	.....
	25 grains.	Bad, lumpy.	3.90 / 4.00 — 1.82
			4.20 / ? mud / 3.90 — 1.83
Potassium bi-chromate aud corrosive sublimate, 16:2.	10 grains.	Passable.	4.20 / 4.20 — 1.82
			4.35 / 4.35 — 1.83
	15 grains.	Fair.	4.20 / 4.25 — 1.82
			4.30 / 4.35 — 1.83
	25 grains.	Good.	4.25 / 4.25 — 1.82
			? mud / ? mud — 1.83*
Potassium bi-chromate and corrosive sublimate, 16:4.	10 grains.	Good.	4.10 / 4.20 — 1.82
			4.40 mud / 4.30 — 1.83
	15 grains.	Good.	4.20 / 4.20 — 1.82
			4.35 / 4.20 — 1.83
	25 grains.	Good.	4.30 / 4.20 — 1.82
			4.20 / ? mud — 1.83*
Potassium bi-chromate and corrosive sublimate, 16:8.	10 grains.	Too flecky.	4.10 / 4.20 — 1.82
			4.20 / 4.30 — 1.83
	15 grains.	Little flecky.	4.20 / 4.25 — 1.82
			4.25 / 4.35 — 1.83
	25 grains.	Little flecky	4.30 / 4.35 — 1.82
			4.40 mud / 4.20 — 1.83*

*Afternoon, room warm.

SUMMARY.

1. The most efficient *single* preservative yet found for composite samples is mercuric chloride (corrosive sublimate); used in quantities of fifteen to twenty-five grains it serves well for monthly periods—the daily samples being 15 c. c., about.

2. Potasstum bi-chromate, in amounts of fifteen or twenty grains, serves well for two week periods, but for monthly periods it is not efficient even in doses of forty grains; larger quantities may prove efficient. In insufficient doses potassium bi-chromate frequently promotes the destruction of the milk-fat, by inducing or favoring a peculiar fermentation, which usually sets in during the third or fourth week and which may be recognized by the fruity odor that is developed.

3. The most efficient *mixture* yet found for *preserving* milk samples, and *perhaps* the best compound all in all for practical use at the creamery, is a mixture of potassium bi-chromate and mercuric chloride (corrosive sublimate), in the proportions of either sixteen to two or sixteen to four, by weight. Of the mixture in proportions of sixteen to two, fifteen to twenty-five grains suffice well for monthly composites; of the sixteen to four mixture, ten to fifteen grains. It is by no means certain that in practical use these mixtures, or either one of them, will prove superior to corrosive sublimate (plus color), but in laboratory experiments they have done so well that the writer feels justified in recommending them for trial by creamerymen.

4. Monthly composites, kept by any preservative yet found, tend frequently to yield results (by the Babcock test) a trifle below the true average of the daily samples, especially if the conditions of temperature and strength of acid are not exactly right; the error, however, under ordinary conditions of practice, seldom exceeds $\frac{1}{10}$ per cent, is frequently less, and with conditions just right seems to be *nil.*

5. Monthly composites preserved with bi-chromate, or mixtures containing bi-chromate, are much inclined to yield black mud in the tests (with the Babcock) if the temperature at testing is too high or the acid too strong. Ordinary variations in temperature—as between morning and afternoon of

an autumn day—exert fully as much influence upon the results of the test as does the exact strength of the acid,—between 1.82 and 1.83 sp. gr. With a temperature not too much above 60 degrees Fahrenheit, a strength (or amount) of acid that *almost* brings black mud in the test, but does not quite, seems the best for obtaining *full* results; in other words, the charge of acid should be as large, or the acid as strong, as may be without producing black mud in the tests. With the pure corrosive sublimate preservatives (plus color for danger signal), there is no trouble with muddy tests.

6. For monthly composites, quart jars are much preferable to pints; the Lightning jar is the most convenient kind the writer has tried.